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SPECIFICATION FOR SODIUM BROMIDE, PURE

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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Indian Standard
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Indian Standard
SPECIFICATION FOR SODIUM BROMIDE, PURE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 June 1964, after the draft finalized by the Alkalis and Chlorine Sectional Committee had been approved by the Chemical Division Council.

0.2 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of test for sodium bromide, pure. The material is used in the chemical industry and the laboratory for the preparation of bromides and in medicine.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of small colourless transparent or opaque crystals, or a white granular powder, free from foreign matter and visible impurities. It is very deliquescent and should be kept in well-closed containers.

2.2 The material shall comply with the requirements given in Table 1 when tested according to the methods specified in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

---

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR SODIUM BROMIDE, PURE

(*Clause* 2.2)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Sodium bromide, percent by weight of dried material, *Min*	98·5	A-2
ii)	Chlorides (as Cl), percent by weight, *Max*	0·6	A-3
iii)	Bromates (as BrO_3), percent by weight, *Max*	0·001	A-4
iv)	Heavy metals (as Pb), percent by weight, *Max*	0·001	A-5
v)	Iron (as Fe), percent by weight, *Max*	0·008	A-6
vi)	Sulphates (as SO_4), percent by weight, *Max*	0·06	A-7
vii)	Moisture, percent by weight, *Max*	5·0	A-8
viii)	Arsenic (as As_2O_3), parts per million, *Max*	10	A-9
ix)	Alkali (as Na_2CO_3), percent by weight, *Max*	0·02	A-10
x)	Barium	To pass test	A-11

3. PACKING AND MARKING

3.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean galvanized iron drums lined with polyethylene or in glass bottles.

3.2 Marking — The containers shall be marked with the name of the material; name of the manufacturer and recognized trade-mark, if any; weight of the material in the container; batch number and the date of manufacture.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

ANALYSIS OF SODIUM BROMIDE, PURE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF SODIUM BROMIDE

A-2.1 Reagents

A-2.1.1 *Standard Silver Nitrate Solution* — 0·1 N.

A-2.1.2 *Concentrated Nitric Acid* — conforming to IS : 264-1950†.

A-2.1.3 *Nitrobenzene*

A-2.1.4 *Ferric Alum Indicator Solution* — saturated solution.

A-2.1.5 *Standard Ammonium Thiocyanate Solution* — 0·1 N.

A-2.2 Procedure — Dry the material to constant weight at 105° ± 2°C. Weigh accurately about 0·4 g of the dried material and dissolve in 50 ml of water. Add with a pipette 50 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid, 15 ml of nitrobenzene and finally 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

A-2.3 Calculation

$$\text{Sodium bromide (as NaBr), percent by weight of the dried material} = \frac{1{\cdot}029\,(50 - V)}{W}$$

where

V = volume in ml of standard ammonium thiocyanate solution used in the titration, and

W = weight in g of the dried material taken for the test.

NOTE — Presence of chlorides increases the results for sodium bromide and the results shall therefore be corrected. One gram of chloride (as Cl) is equivalent to 2·899 g of sodium bromide.

---

*Specification for water, distilled quality (*revised*).

†Specification for nitric acid.

A-3. TEST FOR CHLORIDES

A-3.1 Apparatus

A-3.1.1 *Nessler Tubes* — 50 ml capacity.

A-3.2 Reagents

A-3.2.1 *Dilute Nitric Acid* — 1 : 2.

A-3.2.2 *Hydrogen Peroxide* — Dilute one volume of 30 percent hydrogen peroxide with one volume of water.

A-3.2.3 *Concentrated Nitric Acid* — same as in **A-2.1.2**.

A-3.2.4 *Silver Nitrate Solution* — 10 percent.

A-3.2.5 *Standard Chloride Solution* — Dissolve 0·164 9 g of sodium chloride in water and make up the volume to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of the solution contains 0·01 mg of chloride (as Cl).

A-3.3 Procedure — Dissolve 0·500 g of the material in 15 ml of dilute nitric acid in a small conical flask. Add 6 ml of hydrogen peroxide and digest on a steam-bath until the solution is colourless. Wash down the sides of the flask with water, digest again for 15 minutes, cool and dilute to 250 ml with water. Pipette out 10 ml of the solution into a Nessler tube, dilute to about 25 ml with water, add 1 ml of concentrated nitric acid and 0·5 ml of silver nitrate solution. Dilute to 50 ml and mix well. Carry out a control test in another Nessler tube using 12 ml of standard chloride solution and the same quantities of the other reagents.

A-3.3.1 The material shall be taken as not having exceeded the limit given in Table 1 if the turbidity produced with the material is not greater than that produced in the control test.

A-4. TEST FOR BROMATES

A-4.1 Reagents

A-4.1.1 *Potassium Iodide Solution* — 10 percent.

A-4.1.2 *Starch Solution*

A-4.1.3 *Dilute Sulphuric Acid* — 1 : 15.

A-4.2 Procedure — Dissolve 1·0 g of the material in 10 ml of water. Add 2 drops of potassium iodide solution, 1 ml of starch solution and 5 drops of dilute sulphuric acid. Mix thoroughly and allow to stand for 10 minutes.

A-4.2.1 The material shall be taken as not having exceeded the limit prescribed in Table 1 if no blue colour is produced.

A-5. TEST FOR HEAVY METALS

A-5.1 Apparatus

A-5.1.1 *Nessler Tubes* — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 *p-Nitrophenol Indicator Solution* — Dissolve 0·2 g of *p*-nitrophenol in hot water and dilute to 100 ml.

A-5.2.2 *Dilute Ammonium Hydroxide* — 1 : 9.

A-5.2.3 *Dilute Hydrochloric Acid* — 1 : 99.

A-5.2.4 *Hydrogen Sulphide Solution* — saturated and freshly prepared.

A-5.2.5 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-5.3 Procedure — Dissolve 2·00 g of the material in 25 ml of water in a Nessler tube and add one drop of *p*-nitrophenol indicator solution. Add dilute ammonium hydroxide dropwise until the solution turns yellow. Add dilute hydrochloric acid dropwise until the solution becomes colourless and then add 0·5 ml of the acid in excess. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test in a similar manner in another Nessler tube using 2 ml of standard lead solution.

A-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-6. TEST FOR IRON

A-6.1 Apparatus

A-6.1.1 *Nessler Tubes* — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 *p-Nitrophenol Indicator Solution* — same as in **A-5.2.1**.

A-6.2.2 *Dilute Ammonium Hydroxide* — 1 : 9.

A-6.2.3 *Dilute Hydrochloric Acid* — 1 : 99.

A-6.2.4 *Acetate Buffer Solution* — Add 23 g of anhydrous sodium acetate to 58 ml of acetic acid (2 M) and dilute to one litre with water. Adjust the final *p*H of the solution to 5·0 ± 0·1 with acetic acid or sodium hydroxide solution (10 percent).

A-6.2.5 *o-Phenanthroline Reagent* — Thoroughly mix equal volumes of *o*-phenanthroline solution (0·1 percent aqueous), hydroxylamine hydrochloride solution (10 percent aqueous) and acetate buffer solution.

A-6.2.6 *Standard Iron Solution* — Dissolve 0·702 g of ammonium ferrous sulphate in about 100 ml of water containing 10 ml of dilute sulphuric acid (4 N) and dilute to 1 000 ml. Further, dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0·01 mg of iron (as Fe).

A-6.3 Procedure — Dissolve 1·000 g of the material in 25 ml of water in a Nessler tube and add one drop of *p*-nitrophenol indicator. Add dilute ammonium hydroxide dropwise until the solution turns yellow. Add dilute hydrochloric acid dropwise until the solution becomes colourless and then add 2 ml in excess. Add 5 ml of *o*-phenanthroline reagent, mix well and let stand for 10 minutes. Dilute to 50 ml and mix well. Simultaneously, carry out a control test using 8 ml of standard iron solution.

A-6.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-7. TEST FOR SULPHATES

A-7.1 Apparatus

A-7.1.1 *Nessler Tubes* — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Standard Sulphate Solution* — Dissolve 0·148 g of anhydrous sodium sulphate in water and dilute to one litre in a volumetric flask. One millilitre of the solution contains 0·1 mg of sulphates (as SO_4).

A-7.2.2 *Dilute Hydrochloric Acid* — 1 : 99.

A-7.2.3 *Barium Chloride Solution* — 10 percent.

A-7.3 Procedure — Dissolve 1·000 g of the material in 25 ml of water and transfer to a Nessler tube. In another Nessler tube, carry out a control test using 6 ml of standard sulphate solution and 25 ml of water. To each tube, add 1 ml of dilute hydrochloric acid and 5 ml of barium chloride solution. Dilute the contents of each tube to the mark and stir well.

A-7.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than that produced in the control test.

A-8. DETERMINATION OF MOISTURE

A-8.1 Procedure — Weigh accurately about 5 g of the material in a low-form glass-stoppered weighing bottle. Dry at 105°±2°C for four hours, cool in a desiccator and weigh.

A-8.2 Calculation

$$\text{Moisture, percent by weight} = \frac{100\, W_1}{W_2}$$

where ,

W_1 = loss in weight in g on drying, and

W_2 = weight in g of the material taken for the test.

A-9. TEST FOR ARSENIC

A-9.1 Dissolve 1·000 g of the material in 10 ml of water and carry out the test for arsenic as directed in IS:2088-1962*, using 0·01 mg of arsenic trioxide for preparing the comparison stain.

A-10. TEST FOR ALKALI

A-10.1 Reagents

A-10.1.1 *Phenolphthalein Indicator Solution* — Dissolve 0·1 g of phenolphthalein in 60 ml of rectified spirit conforming to IS : 323-1959† and dilute with water to 100 ml.

A-10.1.2 *Standard Hydrochloric Acid* — 0·02 N.

A-10.2 Procedure — Dissolve 1·00 g of the material in 50 ml of freshly boiled and cooled water and add 5 drops of phenolphthalein indicator solution. If any pink colour is produced, titrate with standard hydrochloric acid.

A-10.2.1 The material shall be taken not to have exceeded the limit prescribed in Table 1 if not more than 0·2 ml of standard hydrochloric acid is required to discharge the pink colour.

A-11. TEST FOR BARIUM

A-11.1 Reagent

A-11.1.1 *Dilute Sulphuric Acid* — approximately 4 N.

A-11.2 Procedure — Dissolve 0·5 g of the material in 10 ml of water and add 1 ml of dilute sulphuric acid.

A-11.2.1 The material shall be taken to have passed the test if no turbidity is produced within 5 minutes.

---

*Modified Gutzeit method of test for arsenic.

†Specification for rectified spirit (*revised*).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF SODIUM BROMIDE, PURE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 The sampling instrument and the containers for the samples shall be such that the material has no action on them. They shall be clean and dry.

B-1.2 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and, as far as possible, the material shall also be protected from light.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment of the material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of the material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 and above	10

NOTE — In the case of very small lots where the selection of 3 containers may be uneconomical, the method of judging the conformity of the lot to the requirements of the specification shall be as agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection, random number tables shall be used. In case these tables are not available, the following procedure is recommended for use:

> Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3,......, up to *r* and so on, where *r* is the integral part of *N*/*n*. Every *r*th container thus counted shall be withdrawn to give sample for tests.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a small representative portion of the material of about 100 g shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions, equal quantities of the material (not more than 90 g) shall be taken and mixed thoroughly to form a composite sample of about 250 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Test for the determination of sodium bromide shall be performed on each of the individual samples (*see* **B-3.3**).

B-4.2 Tests for the determination of all other characteristics given in Table 1 shall be performed on the composite sample (*see* **B-3.2**).

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results for sodium bromide, the mean ($\bar{x}$) and range (R) of test results shall be computed

(range being defined as the difference between the maximum and minimum values of test results).

B-5.1.1 The lot shall be declared as conforming to the requirement for sodium bromide if the value of the expression ($\bar{x} - 0{\cdot}6\ R$) as calculated from the test results is equal to or greater than 98·5.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified.

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